MECHANISM OF IMAGING OF CARBON NANOTUBES BY SCANNING ELECTRON MICROSCOPY

Dmitriy A. Dikin, Rodney S. Ruoff

Department of Mechanical Engineering, Northwestern University, Evanston, IL 60208, USA

Keywords: carbon nanotube, SEM, secondary electrons.

Scanning electron microscopy (SEM) is used for imaging of nanostructures, including carbon nanotubes (CNT). Recent developments of the SEM technique[1,2] mean new capabilities for nanoscale sample analysis with potential industrial and metrological applications. This is further discussed in recent publications,[3,4] however the physics of CNT imaging is not fully understood. We have found interesting physical and chemical phenomena in our "SEM experiments", which can be used for analysis of the interaction between CNTs and neighboring material (such as a substrate). We have analysed the role of the electron beam parameters, the effect of surrounding materials and the substrate, and the influence of the properties of the CNTs on their appearance in SEM micrographs.

We use a LEO 1525 Schottky field emission SEM, which has two secondary-electron detectors. One is a phosphor scintillator detector sitting in the chamber ("side-view") and the second is in the column primary-electron beam ("in-column"). The in-column detector collects low-energy secondary electrons much more efficiently then the side-view detector, which we find at times to be a key advantage of this instrument.

We have studied CNTs grown by a variety of methods in different laboratories: chemical vapour-deposited (CVD) in the Ruoff Lab (Northwestern University), the Liu Lab (Duke University), and the Ren Lab (Boston College), and arc-discharge grown in the Chang Lab (Northwestern University).

In serious of SEM experiments, a variety of sample types have been studied: (i) CNTs in a liquid suspension were spun onto Si substrates with a range of thicknesses of the top oxide layer (2 nm – 1 μm); (ii) as grown CNTs on different substrates—that is, they are attached to the substrate at the synthesis step, and we have studied them as such; (iii) CNTs in contact with metal films. As grown, and also chemically treated, CNTs dispersed in nonconductive polymers were also imaged and their interaction with the surrounding polymer matrix was analysed.

From these imaging studies, some conclusions have been reached: (a) The chemical and physical interaction with the surrounding material and its electrical properties determine the "appearance" of the CNT (Figure 1). (b) By tuning both the primary electron energy and flux it is possible tune the "brightness" of the CNTs (Figure 2). Studies varying such parameters as in (a) and (b) can be used to understand the physics and chemistry of the CNT-surrounding interaction, and interface properties.

SEM was done at the Electron Probe Instrumentation Centre at Northwestern University. Electron beam lithography was done in the Chandrasekhar lab by Zhengfan Zhang (Dept of Physics, Northwestern University). Funding for this work has been provided by a NASA/MSFC Phase II SBIR, Contract No. NAS8-02102, through a subcontract from Lytec, LLC., and by the NSF NIRT Program, Grant No. 0304506.

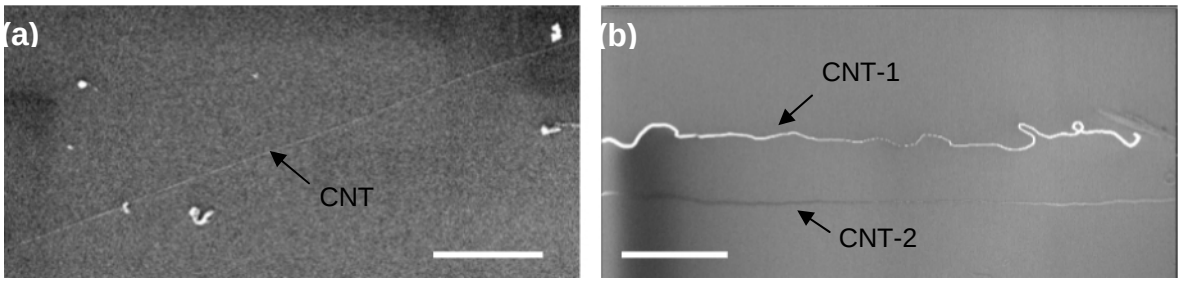

***Figure* 1.** Secondary electron images of CNTs: (a) spun on Si substrate and obtained with the *in-column* detector (3 keV incident electron energy; scale bar is 1 μm); (b) as-grown (CVD) on a Si substrate and obtained by "mixing" the *in-column* and *side-view* detectors in a 0.65:0.35 ratio (1 keV incident electron energy, scale bar is 25 μm).

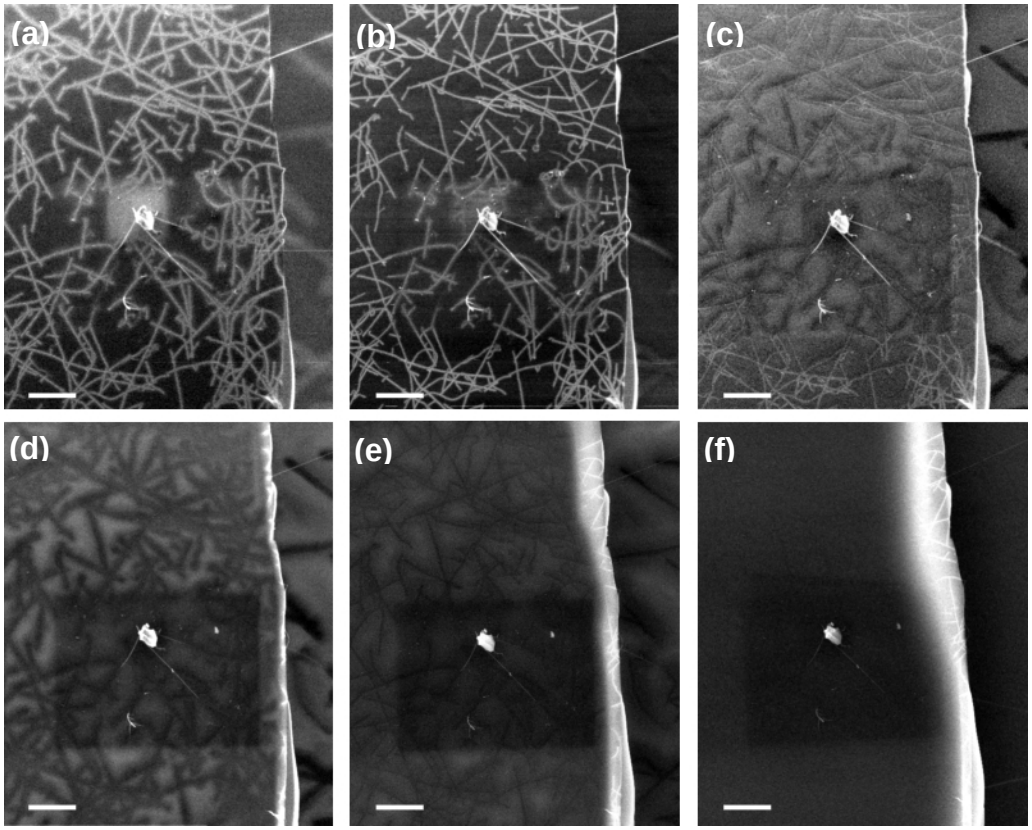

Figure 2. Secondary electron images of CVD-grown CNTs on Si obtained with the *in-column* detector at these incident beam electron energies: (a) 0.3 keV, (b) 0.6 keV, (c) 1.25 keV, (d) 2.5 keV, (e) 5.0 keV, and (f) 10 keV. Scale bar for all images 1 μm.

REFERENCES

1. Jaksch, H.; Martin, J.P.: "High-resolution, low-voltage SEM fro true surface imaging and analysis," *Fresenius J. Anal. Chem.*, **353**, 378-382 (1995).
2. Frank, L.; Zadražil, M.; Müllerova I.: "Scanning electron microscopy of nonconductive specimens at critical energies in a cathode lens system," *Scanning*, **23**, 36-50 (2001).
3. Brintlinger, T., et al., "Rapid imaging of nanotubes on insulating substrates," *Appl. Phys. Lett.*, **81**, 2454-2456 (2002).
4. Homma, Y., et al., "Mechanism of bright selective imaging of single-walled carbon nanotubes on insulators by scanning electron microscopy," *Appl. Phys. Lett.*, **84**, 1750-1752 (2004).